

Supporting Information

to

Highly efficient mesoporous polymer supported phosphine-gold(I) complex catalysts for amination of allylic alcohols and intramolecular cyclization

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Experimental Section

Synthesis of FDU-type mesoporous polymers. Aqueous solution of NaOH (0.13 g, 0.65 mmol, 20w%) was added slowly to Phenol (0.61 g, 6.50 mmol) over 10 min with stirring. aqueous solution of formaldehyde (37 wt%, 1.05 g) was added dropwise, and the reaction mixture was stirred at 70 °C for 60 min. After cooling the mixture to room temperature, the pH of the reaction mixture was adjusted to neutral (7.0) using 0.6M HCl solution. Dried under vacuum, then resol precursor was obtained. pluronic 127 (1.00 g) was dissolved in ethanol (20.0 g), then the ethanol (25 mL) solution of above resol precursor was added by stirring for 10 min. The solution was transferred to a dish and the ethanol evaporated at room temperature over 5-8 h to produce a transparent membrane. The membrane was then heated in an oven at 100 °C for 24 h to thermopolymerize the phenolic resins. The products were calcined at 350 °C under nitrogen for 5 h with a temperature increase rate of 1 °Cmin⁻¹.

Synthesis of FDU-CH₂Cl. Under argon atmosphere, anhydrous AlCl₃ (12 g, 90 mmol) was added in portions to a suspension of mesopolymer (3 g) with chloromethyl methyl ether (30 mL) stirred in ice-water bath, then stirred at room temperature for 24 h. A amount of cold water was slowly pour into the resulting mixture, the suspension was filtered and washed repeatedly with distilled water and acetone, then dried under vaccum.

Synthesis of PS-PPh₂AuCl.^[1] To a suspension of triphenylphosphine resin (0.34 g, 0.34 mmol of the phosphine ligand, 100–200 mesh) in CH₂Cl₂ (10 mL) was added a solution of (Me₂S)AuCl(0.05 g, 0.34 mmol) in CH₂Cl₂(5 mL) at 0 °C. After stirring at room temperature for 6 h, the precipitate was filtered off, washed with CH₂Cl₂, and dried under vacuum. The Au loadings were determined to be 16.0 wt% (ICP).

Synthesis of SBA-PPh₂AuCl. 2.0 g of Pluronic P123 was dissolved in 15 g of water and 60 g of 2 M HCl solution with stirring at 35 °C. Then 4.25 g of TEOS was added into that solution with stirring at 35 °C for 20 h. The mixture was aged at 80 °C overnight without stirring. The solid product washed by ethanol, and dried at 80 °C. Calcination was carried out by slowly increasing temperature from room temperature to 500 °C in 8 h and heating at 500 °C for 6 h. SBA-15 was obtained.^[2]

Diphenyl[3-(triethoxysilyl)propyl]phosphine (0.39 g, 1.0mmol) was added to the suspension of SBA-15 (2 g) in toluene (100 mL) and refluxed for 24 h. The mixture was filtered and the precipitate washed by water and ethanol, dried under vacuum at 60 °C. chloro(dimethylsulfide)gold(I) (0.29 g 1mmol) was added to the suspension of above product in toluene(50 mL), stirring at r.t. over night. The mixture was filtered and washed by ethanol, dried under vacuum at 60 °C and SBA-PPh₂AuCl was obtained.^[3] The Au loadings were determined to be 6.17 wt% (ICP).

Characterization of the FDU-type catalysts.

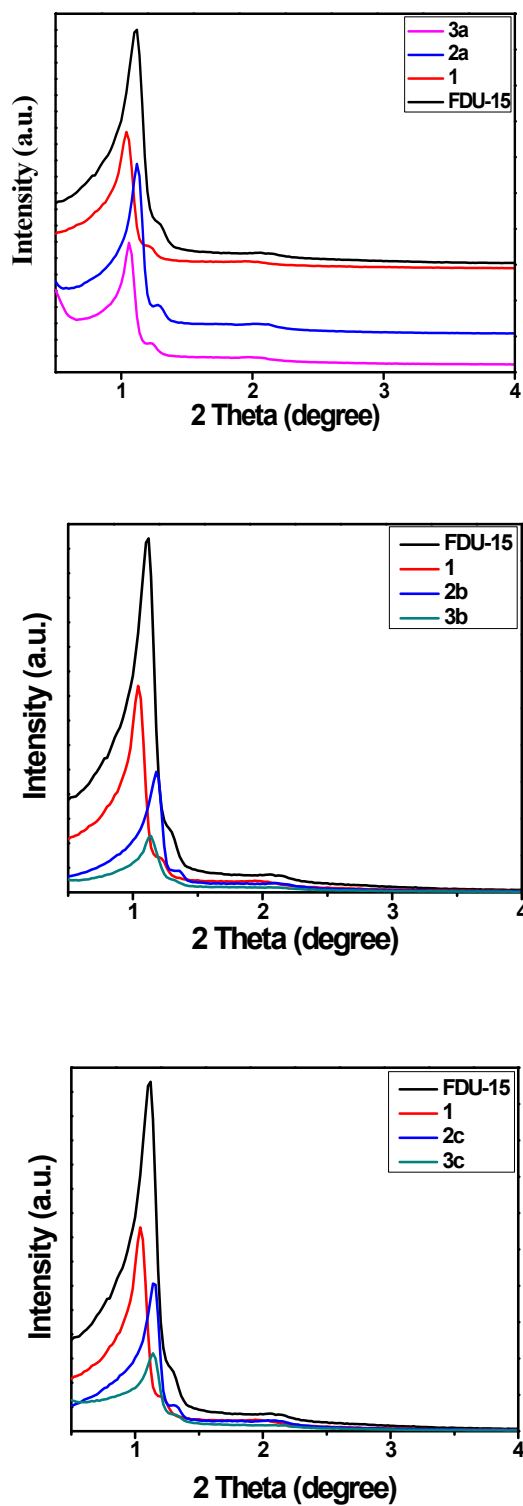


Fig S1. The XRD pattern of catalysts 3a-3c

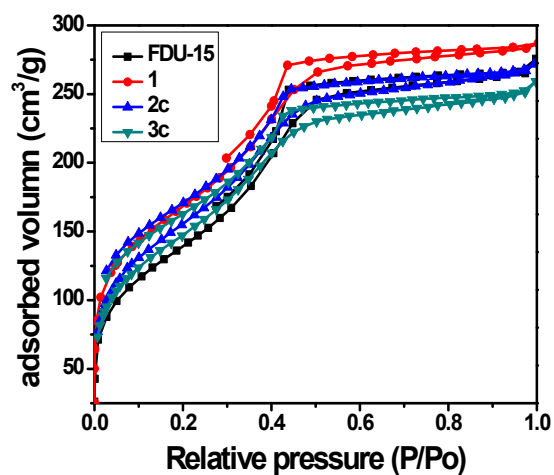
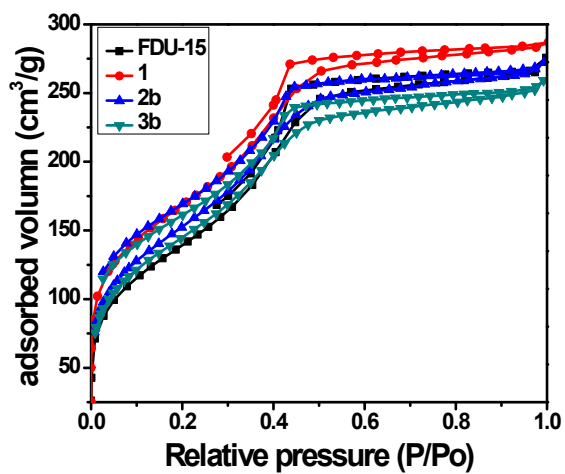
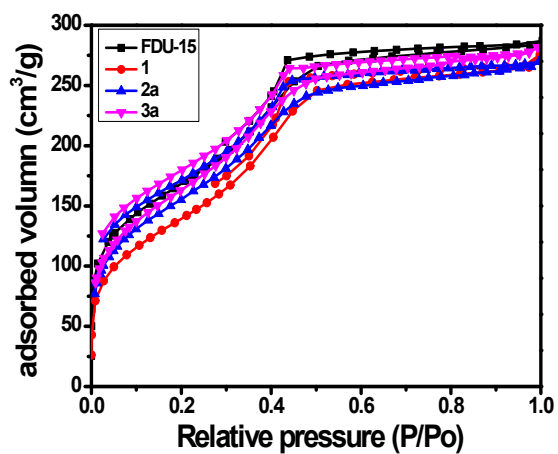


Fig S2. The Nitrogen adsorption–desorption isotherms of catalysts 3a-3c

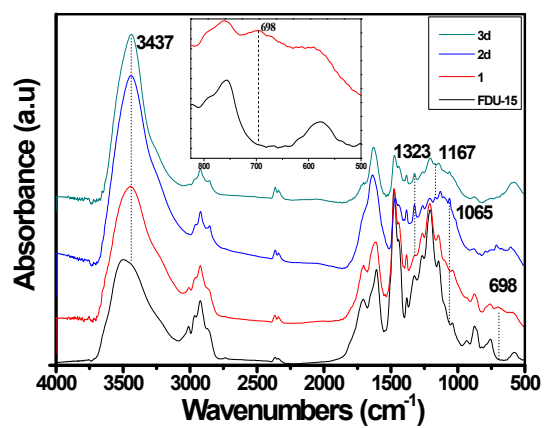
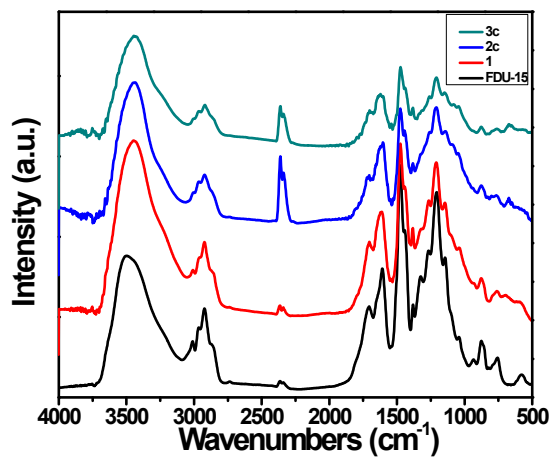
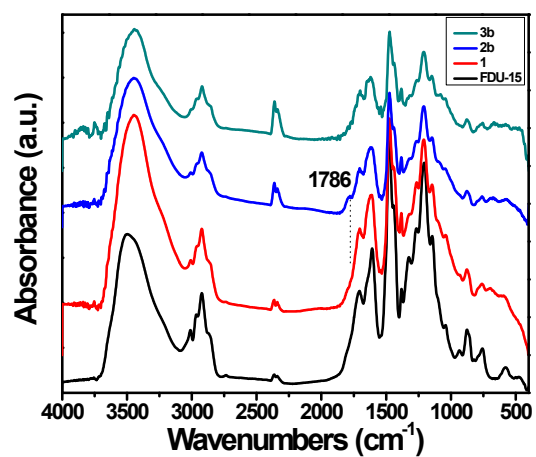


Fig S3. FT-IR spectra of catalysts 3b-3d

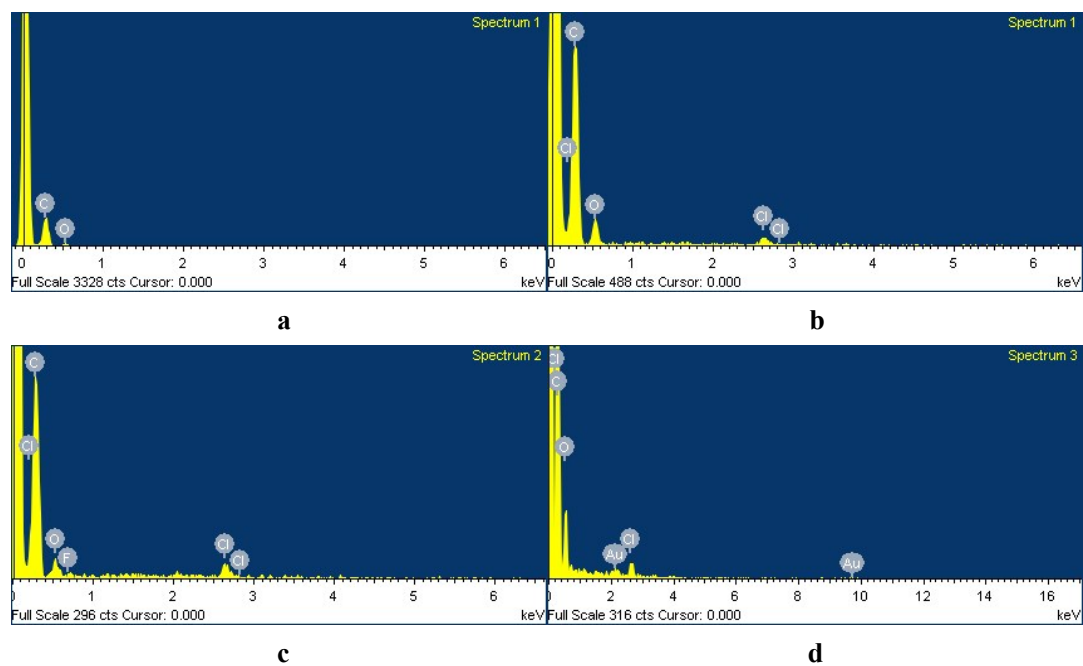


Fig S4. EDS spectra of catalyst a) FDU, b) FDU-Cl, c) FDU-(p-CF₃Ph)₂P and d) 3d

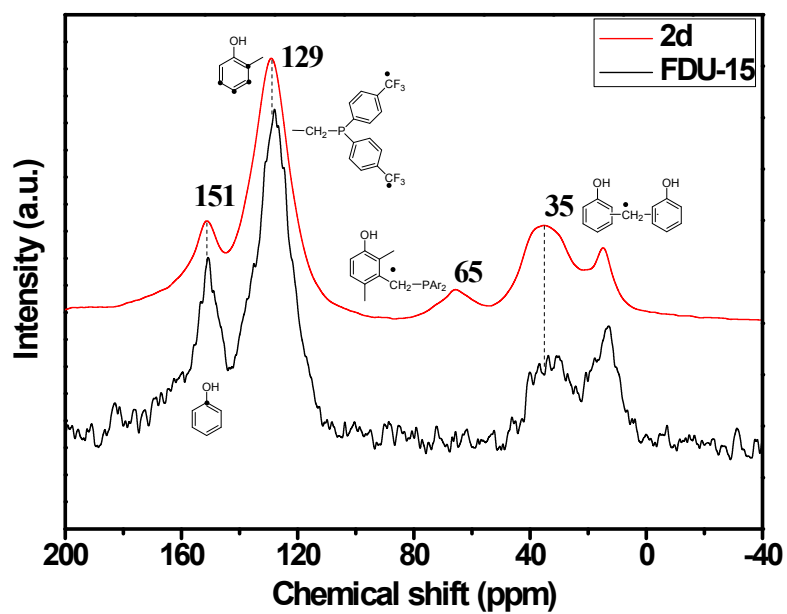


Fig S5. ¹³C MAS NMR spectra of a) FDU-15 and b) 2d

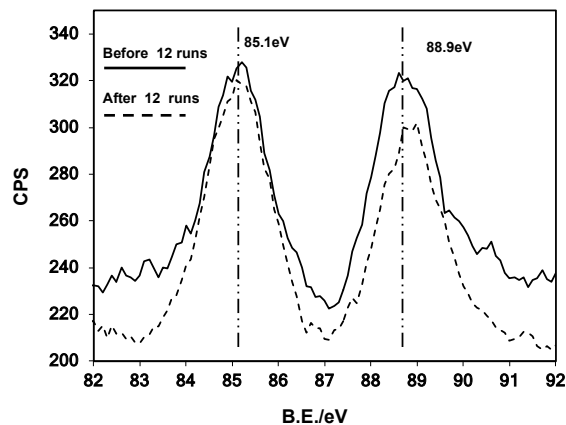


Fig S6. Au 4f XP spectra of sample **3d** before and after 12 runs.

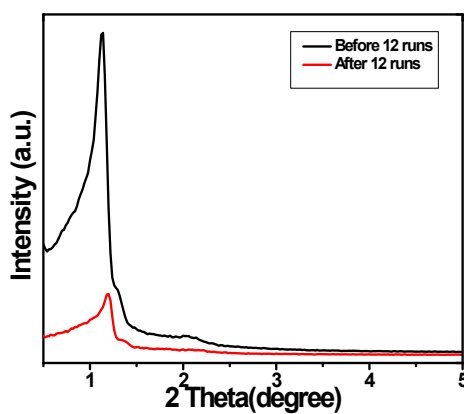


Fig S7. XRD of sample **3d** before and after 12 runs.

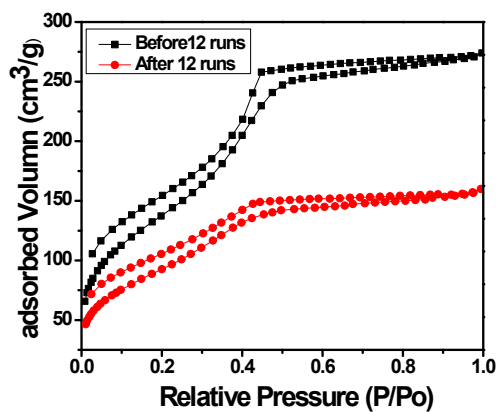


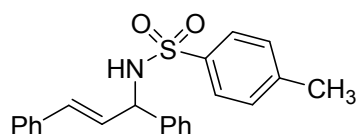
Fig S8. BET of sample **3d** before and after 12 runs.

2. Synthesis of a series of 5-Hydroxy-1-substituted phenyl-2-pentyn-1-one 7a-7l

7a was prepared according to previously reported procedures^[4]. And the substituted **7b-7l** was synthesized by the similar methods. To a solution of 3-butyn-1-ol (0.38 mL, 5.0 mmol) in THF (15 mL) was added EtMgBr (1.0 M in THF; 11mL, 11 mmol) at 0 °C under argon atmosphere, and the reaction was refluxed for 2.5 h. After cooling to -78 °C, a series solution of substituted benzaldehyde (5.0 mmol) in THF (1.5 mL) was added. The mixture was allowed to warm to room temperature, stirred overnight, quenched with sat. aq. NH₄Cl. The organic materials were extracted with EtOAc, and the combined organic extracts were washed with brine, dried over MgSO₄, and evaporated in vacuo. The residue was purified by flash column chromatography and directed used for the next reaction.

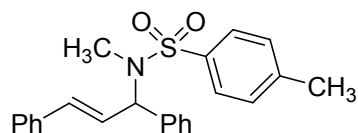
3. ¹H/¹³C NMR and HR-MS Data for substrates

(*E*)-*N*-(1, 3-diphenylallyl)-4-methylbenzenesulfonamide (**6a**)^[5]



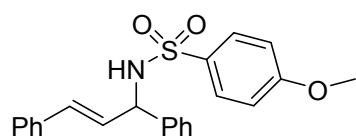
¹H NMR (500 MHz, DMSO-*d*₆) δ 8.35 (d, *J* = 9.0 Hz, 1H), 7.62 (d, *J* = 8.5 Hz, 2H), 7.31 – 7.13 (m, 12H), 6.25 – 6.18 (m, 1H), 6.06 (dd, *J* = 16.0, 7.5 Hz, 1H), 5.01 – 4.97 (m, 1H), 2.24 (s, 3H).

(*E*)-*N*-(1, 3-diphenylallyl)-*N*, 4-dimethylbenzenesulfonamide (**6b**)^[5]



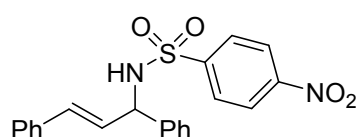
¹H NMR (500 MHz, DMSO-*d*₆) δ 7.71 (d, *J* = 8.5 Hz, 2H), 7.40 – 7.35 (m, 2H), 7.35 – 7.22 (m, 10H), 6.39 (s, 1H), 6.38 (d, *J* = 2.0 Hz, 1H), 5.66 (dd, *J* = 5.0, 2.5 Hz, 1H), 2.64 (s, 3H), 2.27 (s, 3H).

(E)-N-(1, 3-diphenylallyl)-4-methoxybenzenesulfonamide (6c) ^[5]



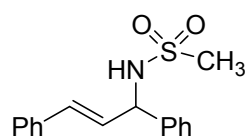
¹H NMR (500 MHz, DMSO-*d*₆) δ 8.28 (d, *J* = 8.0 Hz, 1H), 7.68 – 7.63 (m, 2H), 7.33 – 7.14 (m, 10H), 6.95 – 6.91 (m, 2H), 6.23 (dd, *J* = 16.0, 1.0 Hz, 1H), 6.07 (dd, *J* = 16.0, 7.5 Hz, 1H), 4.97 (t, *J* = 7.5 Hz, 1H), 3.70 (s, 3H).

(E)-N-(1, 3-diphenylallyl)-4-nitrobenzenesulfonamide (6d) ^[5]



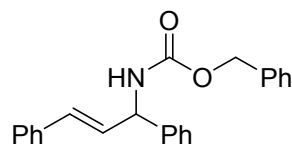
¹H NMR (500 MHz, DMSO-*d*₆) δ 8.84 (d, *J* = 9.0 Hz, 1H), 8.22 – 8.17 (m, 2H), 7.96 – 7.90 (m, 2H), 7.30 – 7.14 (m, 10H), 6.29 (d, *J* = 16.0 Hz, 1H), 6.09 (dd, *J* = 16.0, 7.5 Hz, 1H), 5.10 (t, *J* = 9.0 Hz, 1H).

(E)-N-(1, 3-diphenylallyl) methanesulfonamide (6e) ^[5]



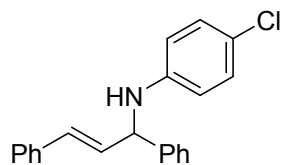
¹H NMR (500 MHz, DMSO-*d*₆) δ 7.97 (d, *J* = 9.0 Hz, 1H), 7.49 – 7.21 (m, 10H), 6.62 (d, *J* = 16.0 Hz, 1H), 6.39 (dd, *J* = 16.0, 7.0 Hz, 1H), 5.14 (t, *J* = 8.5 Hz, 1H), 2.75 (s, 3H).

Benzyl-(E)-(1, 3-diphenylallyl) carbamate (6f) ^[5]



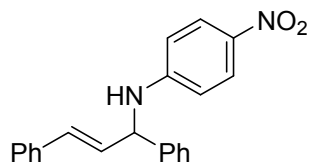
¹H NMR (500 MHz, DMSO-*d*₆) δ 8.14 (d, *J* = 8.5 Hz, 1H), 7.54 – 7.06 (m, 15H), 6.54 (d, *J* = 16.0 Hz, 1H), 6.39 (dd, *J* = 16.0, 6.5 Hz, 1H), 5.39 (t, *J* = 8.5 Hz, 1H), 5.11 – 4.98 (m, 2H).

(E)-4-chloro-N-(1, 3-diphenylallyl) aniline (6g)^[5]



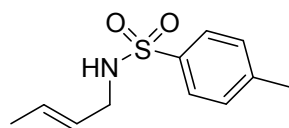
¹H NMR (500 MHz, DMSO-*d*₆) δ 7.47 – 7.18 (m, 10H), 7.04 (d, *J* = 9.0 Hz, 2H), 6.64 (d, *J* = 9.0 Hz, 2H), 6.58 (dd, *J* = 16.0, 1.0 Hz, 1H), 6.51 (d, *J* = 7.0 Hz, 1H), 6.43 (dd, *J* = 16.0, 7.0 Hz, 1H), 5.16 (t, *J* = 7.0 Hz, 1H).

(E)-N-(1, 3-diphenylallyl)-4-nitrobenzenesulfonamide (6h)^[5]



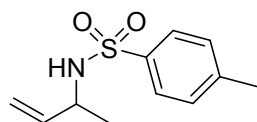
¹H NMR (500 MHz, DMSO-*d*₆) δ 7.98 (d, *J* = 9.5 Hz, 2H), 7.88 (d, *J* = 7.5 Hz, 1H), 7.51 – 7.20 (m, 10H), 6.75 (d, *J* = 9.0 Hz, 2H), 6.62 (d, *J* = 16.0 Hz, 1H), 6.49 (dd, *J* = 16.0, 6.5 Hz, 1H), 5.42 (t, *J* = 6.9 Hz, 1H).

(E)-N-(but-2-en-1-yl)-4-methylbenzenesulfonamide (6i)^[5]



¹H NMR (500 MHz, Chloroform-*d*) δ 7.74 (d, *J* = 8.0 Hz, 2H), 7.31 (d, *J* = 7.5 Hz, 2H), 5.61 – 5.53 (m, 1H), 5.37 – 5.30 (m, 1H), 4.27 (s, 1H), 3.51 (d, *J* = 6.5 Hz, 2H), 2.43 (s, 3H), 1.63 – 1.59 (m, 3H).

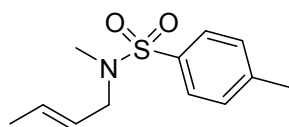
N-(but-3-en-2-yl)-4-methylbenzenesulfonamide (6i')^[5]



¹H NMR (500 MHz, DMSO-*d*₆) δ 7.69 (d, *J* = 8.0 Hz, 1H), 7.67 (d, *J* = 8.5 Hz, 2H), 7.37 (d, *J* = 6.0 Hz, 2H), 5.68 – 5.54 (m, 1H), 4.98 (dt, *J* = 17.0, 1.5 Hz, 1H), 4.87 (dt, *J* = 10.5, 1.0 Hz, 1H), 3.75 – 3.66 (m, 1H), 2.37 (s, 3H), 0.99 (d, *J* = 6.5 Hz, 3H).

(*E*)-*N*-(but-2-en-1-yl)-*N*, 4-dimethylbenzenesulfonamide (6j)^[5]

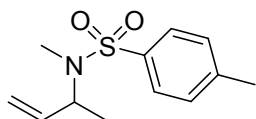
E:*Z* = 17: 2



¹H NMR (500 MHz, DMSO-*d*₆) δ 7.66 (d, *J* = 8.5 Hz, 2H), 7.46 – 7.42 (m, 2H), 5.66 – 5.58 (m, 1H), 5.34 – 5.27 (m, 1H), 3.50 (d, *J* = 6.5 Hz, 2H), 2.55 (s, 3H), 2.41 (s, 3H), 1.63 (dd, *J* = 6.5, 1.5 Hz, 3H).

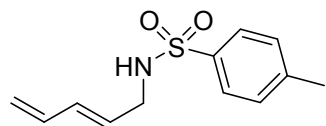
[Distinctive signals for the (*E*)-isomer δ 3.62 (d, *J* = 7.0 Hz, 2H), 1.59 – 1.56 (m, 3H).]

***N*-(but-3-en-2-yl)-*N*, 4-dimethylbenzenesulfonamide (6j')**^[6]



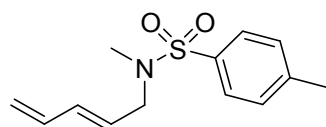
¹H NMR (500 MHz, DMSO-*d*₆) δ 7.69 (d, *J* = 8.0 Hz, 2H), 7.42 (d, *J* = 7.5 Hz, 2H), 5.65 – 5.53 (m, 1H), 5.12 – 5.10 (m, 1H), 5.08 (dt, *J* = 9.0, 1.5 Hz, 1H), 4.54 – 4.47 (m, 1H), 2.58 (s, 3H), 2.40 (s, 3H), 0.98 (d, *J* = 6.5 Hz, 3H).

(*E*)-4-methyl-*N*-(penta-2, 4-dien-1-yl)benzenesulfonamide (6k)^[5]



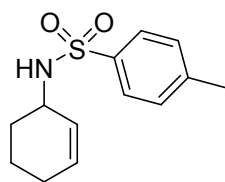
¹H NMR (500 MHz, DMSO-*d*₆) δ 7.71 (t, *J* = 6.0 Hz, 1H), 7.67 (d, *J* = 8.5 Hz, 2H), 7.39 (d, *J* = 8.0 Hz, 2H), 6.25 (dt, *J* = 17.0, 10.0 Hz, 1H), 6.14 – 6.06 (m, 1H), 5.56 (dt, *J* = 15.5, 6.0 Hz, 1H), 5.14 (dd, *J* = 17.0, 2.0 Hz, 1H), 5.05 (dd, *J* = 10.0, 2.0 Hz, 1H), 3.45 – 3.40 (m, 2H), 2.38 (s, 3H).

(*E*)-*N*, 4-dimethyl-*N*-(penta-2, 4-dien-1-yl)benzenesulfonamide (6l)^[7]



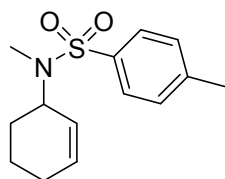
¹H NMR (500 MHz, DMSO-*d*₆) δ 7.68 (d, *J* = 8.0 Hz, 2H), 7.43 – 7.36 (m, 2H), 5.73 – 5.62 (m, 2H), 5.22 – 5.08 (m, 4H), 4.95 – 4.90 (m, 1H), 2.61 (s, 3H), 2.39 (s, 3H).

***N*-(cyclohex-2-en-1-yl)-4-methylbenzenesulfonamide (6m)**^[5]



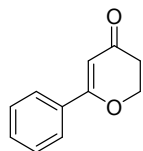
¹H NMR (500 MHz, DMSO-*d*₆) δ 7.79 – 7.62 (m, 3H), 7.45 – 7.30 (m, 2H), 5.73 – 5.62 (m, 1H), 5.30 – 5.20 (m, 1H), 3.64 – 3.56 (m, 1H), 2.38 (s, 3H), 1.92 – 1.78 (m, 2H), 1.64 – 1.53 (m, 2H), 1.44 – 1.31 (m, 2H).

***N*-(cyclohex-2-en-1-yl)-*N*, 4-dimethylbenzenesulfonamide (6n)**^[8]



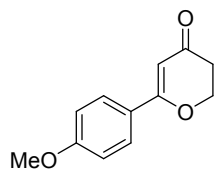
¹H NMR (500 MHz, DMSO-*d*₆) δ 7.69 (d, *J* = 8.0 Hz, 2H), 7.46 – 7.37 (m, 2H), 5.85 – 5.75 (m, 1H), 5.01 – 4.94 (m, 1H), 4.47 – 4.38 (m, 1H), 2.61 (s, 3H), 2.40 (s, 3H), 1.96 – 1.82 (m, 2H), 1.71 – 1.62 (m, 1H), 1.56 – 1.40 (m, 3H).

6-phenyl-2, 3-dihydro-4H-pyran-4-one (8a)^[4]



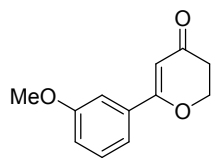
¹H NMR (500 MHz, CDCl₃) δ: 2.67 (2H, t, *J* = 7.0 Hz), 4.67 (2H, t, *J* = 7.0 Hz), 6.03 (1H, s), 7.41–7.52 (3H, m), 7.74 (2H, d, *J* = 7.5 Hz).

6-(4-methoxyphenyl)-2, 3-dihydro-4H-pyran-4-one (8b)



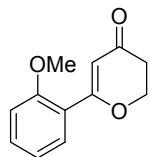
¹H NMR (500 MHz, CDCl₃) δ: 2.67 (2H, t, *J* = 7.25 Hz), 3.89 (3H, s), 4.67 (2H, t, *J* = 7.25 Hz), 5.98 (1H, s), 6.96 (2H, d, *J* = 7.1 Hz), 7.72 (2H, d, *J* = 7.1 Hz); ¹³C MNR (125 MHz, CDCl₃) δ: 36.1, 55.5, 68.1, 101.3, 114.2, 124.9, 128.3, 162.8, 170.6, 192.6. HRMS (EI): *m/z* calcd for C₁₂H₁₂O₃: 204.0786. Found: 204.0788.

6-(3-methoxy-Phenyl)-2, 3-dihydro-4H-pyran-4-one (8c)



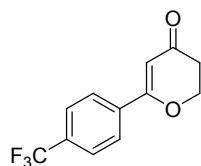
^1H NMR (500 MHz, CDCl_3) δ : 2.68 (2H, t, $J=6.7$ Hz), 3.87 (3H, s), 4.68 (2H, t, $J=6.7$ Hz), 6.03 (1H, s), 7.01-7.08 (1H, m), 7.26-7.28 (1H, m), δ 7.32-7.39 (2H, m); ^{13}C MNR (125 MHz, CDCl_3) δ : 36, 55.4, 68.3, 102.7, 111.6, 117.7, 118.9, 129.7, 134.1, 159.8, 170.3, 192.7. HRMS (EI): m/z calcd for $\text{C}_{12}\text{H}_{12}\text{O}_3$: 204.0786. Found: 204.0783.

6-(2-meoxythyl-Phenyl)-2, 3-dihydro-4H-pyran-4-one (8d)



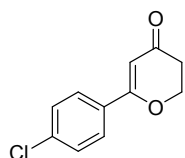
^1H NMR (500 MHz, CDCl_3) δ : 2.68 (2H, t, $J=6.7$ Hz), 3.91 (3H, s), 4.65 (2H, t, $J=6.7$ Hz), 6.33 (1H, s), 7.02 (2H, m), 7.72 (1H, m), 7.45 (1H, m); ^{13}C MNR (125 MHz, CDCl_3) δ : 36, 55.5, 68.1, 107.9, 111.6, 120.7, 129.4, 132.4, 158.2, 168.2, 193.5. HRMS (EI): m/z calcd for $\text{C}_{12}\text{H}_{12}\text{O}_3$: 204.0786. Found: 204.0788.

6-(4-trifluoromethyl-Phenyl)-2, 3-dihydro-4H-pyran-4-one (8e)



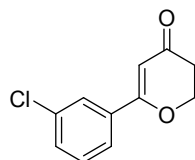
^1H NMR (500 MHz, CDCl_3) δ : 2.72 (2H, t, $J=6.75$ Hz), 4.72 (2H, t, $J=6.75$ Hz), 6.08 (1H, s), 7.72 (2H, d, $J=8.2$ Hz), 7.86 (2H, d, $J=8.2$ Hz); ^{13}C MNR (125 MHz, CDCl_3) δ : 36.1, 68.5, 103.6, 125.7, 126.8, 136, 168.6, 192.4. HRMS (EI): m/z calcd for $\text{C}_{12}\text{H}_9\text{O}_2\text{F}$: 242.0555. Found: 242.0556.

6-(4-chlorine-Phenyl)-2, 3-dihydro-4H-pyran-4-one (8f)



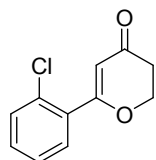
^1H NMR (500 MHz, CDCl_3) δ : 2.69 (2H, t, $J=6.7$ Hz), 4.69 (2H, t, $J=6.7$ Hz), 6.02 (1H, s), 7.43 (2H, d, $J=8.7$ Hz), 7.69 (2H, d, $J=8.7$ Hz); ^{13}C MNR (125 MHz, CDCl_3) δ : 36, 68.3, 102.7, 127.8, 129, 131.1, 137.9, 169.5, 192.4. HRMS (EI): m/z calcd for $\text{C}_{11}\text{H}_9\text{O}_2\text{Cl}$: 208.0291. Found: 208.0290.

6-(3-chlorine-Phenyl)-2, 3-dihydro-4H-pyran-4-one (8g)



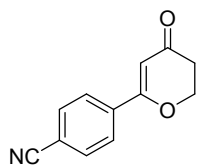
^1H NMR (500 MHz, CDCl_3) δ : 2.69 (2H, t, $J=6.85$ Hz), 4.69 (2H, t, $J=6.85$ Hz), 6.02 (1H, s), 7.46-7.51 (1H, m), 7.67-7.64 (1H, m), 7.72-7.78 (1H, m); ^{13}C MNR (125 MHz, CDCl_3) δ : 36, 68.4, 103.1, 124.5, 126.6, 129.9, 131.5, 134.9, 168.8, 192.4. HRMS (EI): m/z calcd for $\text{C}_{11}\text{H}_9\text{O}_2\text{Cl}$: 208.0291. Found: 208.0292.

6-(2-chlorine-Phenyl)-2, 3-dihydro-4H-pyran-4-one (8h)



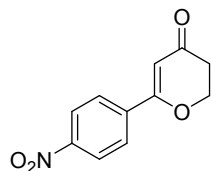
^1H NMR (500 MHz, CDCl_3) δ : 2.71(2H, t, $J=6.85$ Hz), 4.70 (2H, t, $J=6.85$ Hz), 5.79 (1H, s), 7.31-7.43 (2H, m), 7.44-7.51 (2H, m); ^{13}C MNR (125 MHz, CDCl_3) δ : 36, 68.6, 107.9, 126.8, 130.2, 131.6, 133, 170.5, 192.3. HRMS (EI): m/z calcd for $\text{C}_{11}\text{H}_9\text{O}_2\text{Cl}$: 208.0291. Found: 208.0292.

6-(4-cyano-Phenyl)-2, 3-dihydro-4H-pyran-4-one (8i)



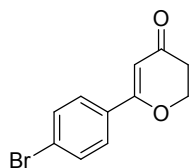
^1H NMR (500 MHz, CDCl_3) δ : 2.72 (2H, t, $J=6.7$ Hz), 4.72 (2H, t, $J=6.7$ Hz), 6.08 (1H, s), 7.75 (2H, d, $J=8.5$ Hz), 7.86 (2H, d, $J=8.5$ Hz); ^{13}C MNR (125 MHz, CDCl_3) δ : 36.1, 68.6, 104.2, 115, 118.1, 126.9, 132.4, 136.9, 168, 192.1. HRMS (EI): m/z calcd for $\text{C}_{12}\text{H}_9\text{O}_2\text{N}$: 199.0633. Found: 199.0635.

6-(4-nitro-Phenyl)-2, 3-dihydro-4H-pyran-4-one (8j)



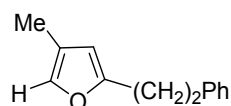
^1H NMR (500 MHz, CDCl_3) δ : 2.73 (2H, t, $J=6.7$ Hz), 4.75 (2H, t, $J=6.7$ Hz), 6.12 (1H, s), 7.89-7.96 (2H, m, $J=8.5$ Hz), 8.28-8.34 (2H, m, $J=8.5$ Hz); ^{13}C MNR (125 MHz, CDCl_3) δ : 36, 68.7, 104.6, 123.8, 127.3, 138.6, 149.5, 167.5, 192.2. HRMS (EI): m/z calcd for $\text{C}_{11}\text{H}_9\text{O}_4\text{N}$: 219.0532. Found: 219.0529.

6-(4-bromine-Phenyl)-2, 3-dihydro-4H-pyran-4-one (8k)



^1H NMR (500 MHz, CDCl_3) δ : 2.68 (2H, t, $J=6.85$ Hz), 4.69 (2H, t, $J=6.85$ Hz), 6.02 (1H, s), 7.65-7.54 (4H, m, $J=8.5$ Hz), 8.28-8.34 (2H, m, $J=8.5$ Hz); ^{13}C MNR (125 MHz, CDCl_3) δ : 36, 68.3, 102.6, 126.3, 127.9, 131.6, 131.9, 169.3, 192.4. HRMS (EI): m/z calcd for $\text{C}_{11}\text{H}_9\text{O}_2\text{Br}$: 251.9786. Found: 251.9788.

4-Methyl-2-(2-phenylethyl) furan (10)^[9]



¹H NMR (500 MHz, CDCl₃) δ: 1.87 (3H, s), 2.75-2.87 (4H, m), 5.75 (1H, s), 6.98 (1H, s), 7.07-7.20 (5H, m); ¹³C MNR (125 MHz, CDCl₃) δ: 9.85, 30.25, 34.54, 108.09, 120.55, 126.12, 128.40, 128.42, 137.49, 141.41, 155.50.

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